



LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

DiMES Studies of Temperature Dependence of Carbon Erosion and Re-Deposition in the DIII-D Divertor

D.L. Rudakov, W. Jacob, K. Krieger, A. Litnovsky, V. Philipps, W.P. West, C.P.C. Wong, S.L. Allen, R.J. Bastasz, J.A. Boedo, N.H. Brooks, R.L. Boivin, G. De Temmerman, M.E. Fenstermacher, M. Groth, E.M. Hollmann, C.J. Lasnier, A.G. McLean, R.A. Moyer, P.C. Stangeby, W.R. Wampler, J.G. Watkins, P. Wienhold, J. Whaley

October 4, 2006

Physica Scripta

Disclaimer

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

DiMES studies of temperature dependence of carbon erosion and re-deposition in the DIII-D divertor

D.L. Rudakov,^a W. Jacob,^b K. Krieger,^b A. Litnovsky,^c V. Philipps,^c W.P. West,^d C.P.C. Wong,^d S.L. Allen,^e R.J. Bastasz,^f J.A. Boedo,^a N.H. Brooks,^d R.L. Boivin,^d G. De Temmerman,^g M.E. Fenstermacher,^e M. Groth,^e E.M. Hollmann,^a C.J. Lasnier,^e A.G. McLean,^h R.A. Moyer,^a P.C. Stangeby,^h W.R. Wampler,ⁱ J.G. Watkins,ⁱ P. Wienhold,^c J. Whaley^f

^a*University of California, San Diego, La Jolla, California 92093-0417, USA*

^b*Max-Planck-Institut für Plasmaphysik, EURATOM Association, 85748 Garching, Germany*

^c*Institut für Plasmaphysik, Forschungszentrum Jülich, Association EURATOM-FZJ, Germany*

^d*General Atomics, San Diego, California 92186-5608, USA*

^e*Lawrence Livermore National Laboratory, Livermore, California, USA*

^f*Sandia National Laboratories, Livermore, California, USA*

^g*Institute of Physics, University of Basel, Klingelbergstrasse 82, CH-4056, Basel, Switzerland*

^h*University of Toronto Institute for Aerospace Studies, Toronto, Ontario, Canada M3H 5T6*

ⁱ*Sandia National Laboratories, Albuquerque, New Mexico 87185, USA*

Abstract

A strong effect of a moderately elevated surface temperature on net carbon deposition and deuterium co-deposition in the DIII-D divertor was observed under detached conditions. A DiMES sample with a gap 2 mm wide and 18 mm deep was exposed to lower-single-null (LSN) L-mode plasmas first at room temperature, and then at 200°C. At the elevated temperature, deuterium co-deposition in the gap was reduced by an order of magnitude. At the plasma-facing surface of the heated sample net carbon erosion was measured at a rate of 3

nm/s, whereas without heating net deposition is normally observed under detachment. In a related experiment three sets of molybdenum mirrors recessed 2 cm below the divertor floor were exposed to identical LSN ELMy H-mode discharges. The first set of mirrors exposed at ambient temperature exhibited net carbon deposition at a rate of up to 3.7 nm/s and suffered a significant drop in reflectivity. In contrast, two other mirror sets exposed at elevated temperatures between 90°C and 175°C exhibited virtually no carbon deposition.

PACS numbers: 52.40.Hf, 52.55.Fa, 52.55.Rk, 81.15.Cd

I. INTRODUCTION

The Divertor Material Evaluation System (DiMES) [1] at the DIII-D tokamak [2] is aimed at studies of plasma-material interactions in a tokamak divertor. The DiMES system allows insertion of material samples into the lower divertor floor, exposing them to either a single plasma discharge, or a series of reproducible discharges to increase the total exposure time. Since DIII-D has plasma-facing components (PFCs) made of carbon, studies of carbon erosion, migration and re-deposition constitute the focus of the DiMES research program.

The current design of the ITER divertor incorporates carbon-fiber-composite tiles at the divertor targets [3]. Carbon will be eroded from the targets by physical and chemical sputtering, and then re-deposited on the PFCs [4]. This process may lead to a number of potential problems for plasma operations, in particular tritium accumulation due to co-deposition with carbon on the faces of PFCs and in gaps between them, and deterioration of diagnostic mirrors from carbon deposition. These issues have been recognized as high priority International Tokamak Physics Activity (ITPA) topics that need to be resolved for ITER to succeed.

Laboratory experiments with thermal ion beams have shown that the chemical erosion rate of carbon peaks at about 400°C, where it is about an order of magnitude higher than at room temperature [5,6]. An exponential increase of the re-erosion rates of plasma-deposited a-C:H layers with increasing substrate temperature has been observed in low-temperature methane plasmas [7,8]. Although pure methane was used as working gas in these experiments, a transition from net deposition at ambient temperature to net erosion at elevated temperature was observed. The transition temperature depends on experimental conditions, such as ion energy and particle fluxes to the surface. It was between 200 and 300°C in these experiments [7]. Similar results, i.e. a transition from net deposition to net erosion, were found in the PSI-2

plasma generator [9]. Under these experimental conditions, the transition temperature was 100°C. There is a qualified hope that increased surface temperature of the PFCs may be used in ITER to mitigate carbon re-deposition and tritium co-deposition in the areas where it is most undesirable. To test this, the capability for in-situ temperature control of the samples was recently implemented in DiMES. Some recent experiments performed at DIII-D to study the temperature dependence of the carbon erosion/re-deposition are reviewed in this article.

II. STUDIES OF CARBON DEPOSITION AND DEUTERIUM CO-DEPOSITION DOWN TILE GAPS

Uncontrolled increase of tritium inventory due to co-deposition with carbon is a critical challenge for ITER [4]. A large fraction of the retained tritium may be accumulated in gaps between PFCs, which are not accessible for most proposed tritium removal techniques. A series of dedicated experiments was performed in DIII-D to measure carbon deposition and deuterium co-deposition (as a proxy for tritium) in a simulated tile gap and an attempt to mitigate the deposition by elevated surface temperature was made.

A graphite DiMES sample head with a simulated tile gap 2 mm wide, 15 mm long and 18 mm deep has been designed and fabricated at Sandia National Laboratory (SNL), Livermore. The inside of the gap was lined with silicon catcher plates to optimize the resolution of the deposition thickness measurements. The sample featured a built-in heater and a thermocouple for in-situ temperature control.

In order to quantify the mitigation of carbon deposition and deuterium co-deposition by chemical re-erosion, two exposures of the tile gap sample were performed, first at room temperature ($\sim 30^\circ\text{C}$) and second at 200°C . In both experiments the samples were exposed to nine reproducible ohmic lower single-null (LSN) discharges in deuterium with the outer strike point (OSP) kept at the DiMES radial location for most of the discharge. The discharge parameters were: toroidal magnetic field, $B_T = 2\text{ T}$, plasma current, $I_p = 1.1\text{ MA}$, ohmic

heating power, $P_{ohm} \sim 1$ MW. The line average density was at $4.5 \times 10^{13} \text{ cm}^{-3}$ for about 3 s, and the OSP was detached most of the time. The total exposure time in both cases was about 30 s. In each case, the sample was inserted so that its plasma-facing surface was leveled with the divertor floor and the gap was oriented radially. Photographs of the plasma-facing surface of the gap sample after each plasma exposure are shown in Fig. 1.

After each exposure, the silicon catcher plates were removed from the gap and shipped to Max-Planck-Institut für Plasmaphysik (Garching, Germany) for analysis of the deposits by ellipsometry and ion beam analysis (IBA). Both ellipsometry and IBA analyses of the catcher plates from the non-heated exposure showed measurable amounts of deposited carbon on all plates. The deposit thickness on the side plates decreased exponentially with the distance from the plasma-facing side of the gap, with a decay length of about 1–3 mm [Fig 2(a)]. The deposit thickness profiles from ellipsometry were in good agreement with the carbon number density from IBA. Measured D/C atomic ratio from IBA was 0.4–0.7.

Figure 2 shows measured profiles of the carbon (a) and deuterium (b) number density versus the distance from the gap entrance for the non-heated and heated exposures. Carbon deposition in the heated gap was lower by a factor of 3-4, and the amount of co-deposited deuterium was reduced by about an order of magnitude in the heated exposure [note the different scale in Fig. 2(b)]. This is a very encouraging result for ITER, suggesting that moderately elevated temperature can significantly reduce tritium accumulation in tile gaps. Ellipsometry analysis of the carbon deposition on the catcher plates from the heated exposure failed to resolve the deposition thickness. This may indicate that the carbon reacted with silicon to form a thin Si:C layer rather than a “normal” a-C:H film. More details on the results of the catcher plate analyses are available in Ref. [10].

No measurements of the net erosion/deposition rate at the plasma-facing surface of the sample were available for the non-heated exposure. However, the colored area seen in figure

1(a) is a strong indication of an a-C:H film deposited on the surface. This is in agreement with earlier studies [11] which show net deposition under detached divertor conditions. For the heated exposure a depth-marked graphite button was installed on the plasma-facing surface of the sample [Fig. 1(b)]. After the exposure the button was analyzed by IBA at SNL Albuquerque. Total net erosion of 90 ± 4 nm, corresponding to an average rate of about 3 nm/s was measured at five different locations on the button. This is a rather high erosion rate (in reactor terms equal to about 9 cm per burn-year), comparable to the highest erosion rates observed in DIII-D divertor under attached high-power H-mode conditions [11]. The most reasonable explanation for such a high erosion rate observed under detachment is enhancement of chemical erosion by the elevated surface temperature, in line with observations of Refs. [7,8]. To test this hypothesis, we exposed another depth marked DiMES sample maintained at ambient temperature to a series of seven L-mode discharges with detached OSP and plasma parameters close to those of the tile-gap-exposure discharges. In this experiment the OSP was swept across the divertor floor radially inward, so that during each discharge DiMES spent about 2 s in the private flux zone (PFZ), then about 1 s near the OSP, and finally about 0.5 s in the scrape-off-layer (SOL) just outside the OSP. The total exposure times at the PFZ, OSP and SOL were about 14, 7 and 3.5 s, respectively. The resulting net erosion/deposition was too low to be definitively resolved by IBA, though within the error bars for the measurement the data were biased towards net deposition (Fig. 3). The high net erosion rate observed in the heated experiment was definitely not reproduced. Therefore, the erosion must have been caused by the elevated surface temperature. We should note that the thermal contact of the button with the rest of the heated sample was not optimized, so the button could be heated by the plasma radiation and impinging plasma fluxes to temperatures significantly above the bulk sample temperature of 200°C measured by the thermocouple.

III. MITIGATION OF CARBON DEPOSITION ON DIAGNOSTIC MIRRORS BY INCREASED SURFACE TEMPERATURE

Optical diagnostics in ITER will rely on plasma-facing mirrors to view the plasma [12]. The mirrors will be used over a wide wavelength range and will be required to maintain good optical performance over prolonged time scales. They may suffer from erosion by energetic plasma particles and charge exchange atoms and deposition of surface contaminants, the latter being of particular concern in the divertor region where the neutral densities are high [12].

First dedicated tests of ITER-candidate molybdenum mirrors were recently performed in the lower divertor of DIII-D using DiMES. Three experiments were performed. In each experiment two mirrors were installed on a specially designed stainless steel holder that was inserted in the divertor floor, as shown in Fig. 4. The two mirrors faced opposite toroidal directions, their centers were about 2 cm below the floor. The mirrors were exposed to highly reproducible ELMing (featuring edge localized modes) H-mode LSN discharges in deuterium with the following discharge parameters: $B_T = 2$ T, $I_p = 1.1$ MA, neutral beam heating power, $P_{\text{NBI}} = 6.6$ MW, average plasma density, $\bar{n}_e = 8 \times 10^{19} \text{ m}^{-3}$. During the exposure the mirrors were located in the private flux zone (PFZ). The divertor was detached and plasma temperature and density in the PFZ near the floor were $T_e = 0.5\text{-}2$ eV and $n_e = 4\text{-}8 \times 10^{20} \text{ m}^{-3}$. The deuteron flow to the divertor floor was in the direction of B shown in Fig. 4 (as confirmed by Mach probe measurements); hence the designations “upstream” and “downstream”.

Two sets of mirrors were exposed on two consecutive days. The first set was exposed to six plasma discharges for a total time of about 25 s. No active temperature control was used, but the holder and the mirrors were heated by the plasma radiation and charge exchange atom

fluxes. At the beginning of the exposure the holder temperature was 23°C. After each exposure discharge the temperature had increased by about 9°C (as measured 30 s after the discharge to avoid pickup from the decaying magnetic fields); it then decreased slowly during the 10 minute interval between shots. By the end of the 6th exposure discharge, the holder temperature was at about 44°C [Fig. 5].

Upon removal from the vacuum chamber, visible deposits were found on both mirrors [Figs. 6(a) and 6(b)]. A strong asymmetry was seen in the deposition between the upstream and downstream mirrors. The deposition on the upstream mirror was rather uniform, while on the downstream mirror a clear gradient was observed, with the heaviest deposits found near the top of the mirror. This was probably caused by re-deposition of the carbon sputtered locally from the leading edge of the downstream graphite tile. The areal carbon coverage, measured by secondary ion mass spectroscopy (SIMS) and IBA, was comparable in the centers of the upstream and downstream mirrors, while at the top of the downstream mirror it was a factor of 2 higher. The thickness of the deposited hydrocarbon film was largest near the top of the downstream mirror; its value of 93 nm measured by spectroscopic ellipsometry corresponds to a net deposition rate of 3.7 nm/s. The D:C ratio in the film was about 0.5, as measured by IBA. The deposited film caused the mirror reflectivity in the wavelength range between 250-1000 nm to degrade by 15-60%.

In order to mitigate the carbon deposition by chemical re-erosion, the mirrors were pre-heated using the internal heater prior to the second exposure. A failure of the heater before the experiment caused the holder and the mirrors to slowly cool down under vacuum during the exposure. The holder temperature at the beginning of the experiment was at 140°C, decreasing to about 90°C by its end [Fig. 5]. The mirrors were exposed to 17 plasma discharges, for a total time of about 70 s.

When the mirrors were removed from DiMES, no deposits were visible on either of them [Figs. 6(c) and 6(d)]. Low carbon content on the heated mirror surfaces was confirmed by SIMS and IBA analyses. The areal carbon density on the heated mirrors from IBA was a factor of 10-30 lower than on the non-heated ones. Since the total exposure time of the heated mirrors was almost three times longer compared to non-heated ones, this corresponds to the net carbon deposition rate being lower by a factor of 30-100. The reflectivity of the heated mirrors was essentially preserved in the wavelength range above 500 nm. Between 250-500 nm the reflectivity was slightly degraded due to a thin (<15 nm) oxide film formed on the mirrors, presumably from long term storage in air.

We should note that the thermal contact between the mirrors and the holder was not optimized, so by the end of each plasma discharge the mirrors could be at a higher temperature than the bulk temperature of the holder, as measured by the thermocouple. More recently another mirror exposure with better temperature measurement and control has been performed. In this experiment the temperature was measured directly at the back of one of the mirrors and maintained at $150 \pm 7^\circ\text{C}$ prior to each plasma discharge. The mirrors were exposed to 8 plasma discharges with parameters similar to those of the previous experiments for a total of about 36 s. The increase of the mirror temperature after each discharge was about 15°C (upper curve in Fig.5, measured 30 s after the discharge). No visible deposition was observed on either of the mirrors after the exposure. Therefore, mitigation of carbon deposition on the mirrors by moderately increased temperature was confirmed. Detailed analysis of the mirrors from the third set will be forthcoming.

IV. SUMMARY

In a number of recent experiments in the lower divertor of DIII-D under detached conditions, a moderately elevated surface temperature has been observed to significantly influence carbon erosion/re-deposition and deuterium co-deposition rates. The effect may be qualitatively explained by increased chemical erosion rates at elevated temperatures [5-9]. The effect is observed on both plasma-facing surfaces and surfaces not accessible to charged particles and appears to affect bulk carbon as well as plasma-deposited a-C:H films. The observed reduction of carbon deposition and hydrogenic co-deposition down tile gaps at elevated temperature is potentially good news for ITER. However, if the carbon erosion rates from the plasma-facing surfaces are also increased at higher temperatures, increased carbon impurity production may offset the advantage of higher re-erosion rates in the gaps. On the other hand, temperature control offers an attractive means of mitigating carbon deposition on small individual objects, such as diagnostic mirrors, that can be heated to moderately high temperatures without negative consequences.

ACKNOWLEDGMENTS

This work was performed and supported in part by the U.S. Department of Energy under DE-FG02-04ER54758, W-7405-ENG-48, DE-FC02-04ER54698, and DE-AC04-94AL85000. The mirror exposures were performed in the framework of bilateral U.S.-EURATOM Exchange program.

REFERENCES

- [1] C.P.C. Wong, *et al.*, J. Nucl. Mater. **258-263**, 433 (1998).
- [2] J.L. Luxon, Nucl. Fusion **42**, 614 (2002).
- [3] R. Aymar, *et al.*, Plasma Phys. Control. Fusion **44**, 519 (2002).
- [4] G. Federici, *et al.*, Nucl. Fusion **41**, 1967 (2001).
- [5] J.W. Davis, *et al.*, J. Nucl. Mater. **155-157**, 234 (1988).
- [6] E. Vietzke, V. Philipps, Fusion Technol. **15**, 108 (1989).
- [7] A. von Keudell and W. Jacob, J. Appl. Phys. **79**, 1092 (1996).
- [8] W. Jacob, J. Nucl. Mater. **337-339**, 839 (2005).
- [9] D. Naujoks, *et al.*, Phys. Scripta **T111**, 80 (2004).
- [10] K. Krieger, *et al.*, “Formation of deuterium-carbon inventories in gaps of plasma facing components,” submitted to J. Nucl. Mater.
- [11] D.G. Whyte, *et al.*, Nucl. Fusion **41**, 1243 (2001).
- [12] V. Voitsenya, *et al.*, Rev. Sci. Instrum. **72**, 475 (2001).

Figure captions

Fig. 1. Plasma-facing surface of the tile-gap sample after non-heated (a) and heated (b) plasma exposures.

Fig. 2. Profiles of the carbon (a) and deuterium (b) deposition versus the distance from the gap entrance for the non-heated (black) and heated (red) exposures. The carbon deposition profile in (a) compares well with the thickness profile measured by ellipsometry (green).

Fig. 3. Profile of the net carbon erosion across a non-heated DiMES sample exposed to seven detached LSN L-mode discharges with OSP sweeps. Negative erosion corresponds to net deposition.

Fig. 4. Geometry of the mirror exposure experiment in DIII-D divertor.

Fig. 5. Time history of the mirror holder temperature for the three mirror exposure experiments. Solid symbols correspond to the temperature measured 15 s before and open symbols 30 s after each exposure discharge.

Fig. 6. Mirrors exposed at ambient (a), and (b) and elevated (c), and (d) temperature.

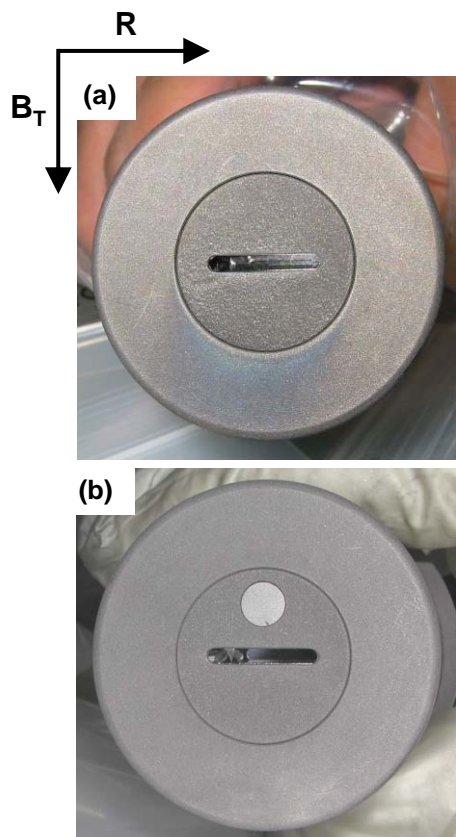


Figure 1

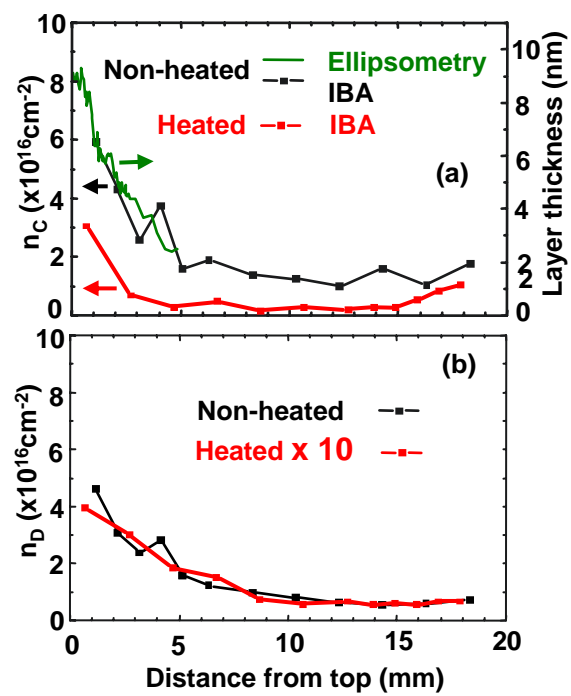


Figure 2

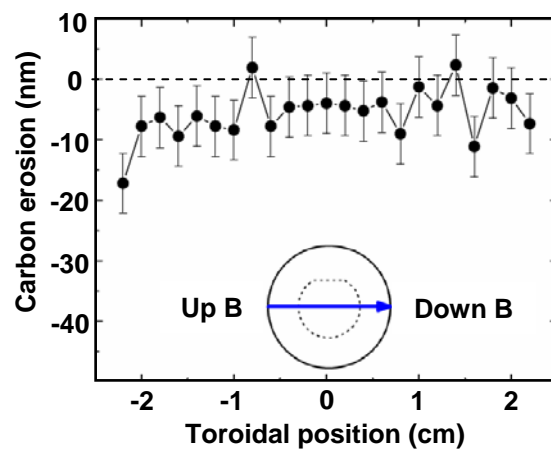


Figure 3

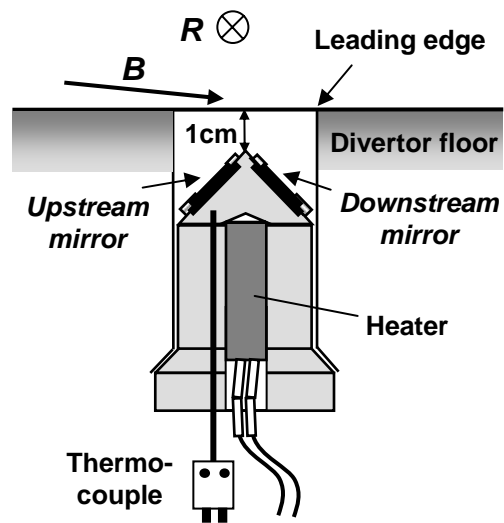


Figure 4

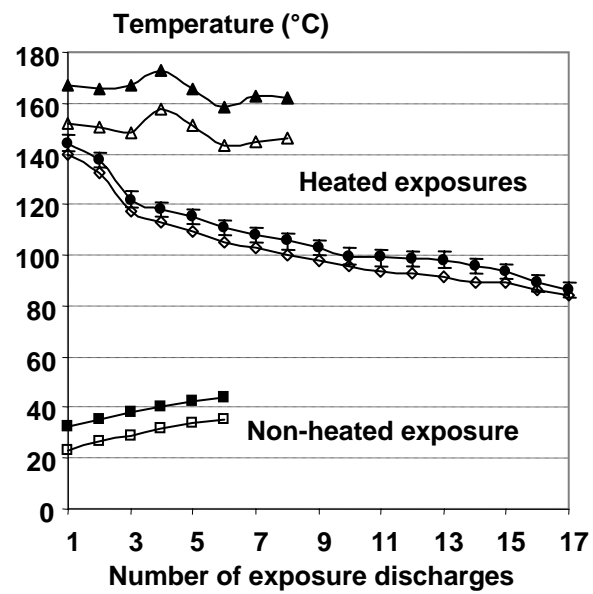


Figure 5

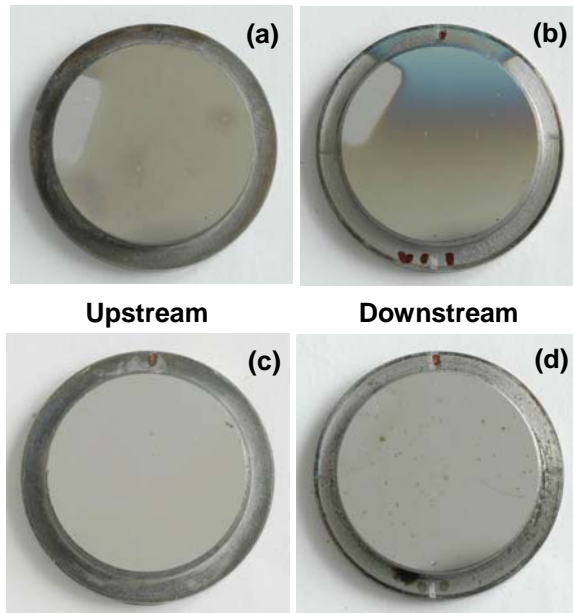


Figure 6